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**EUROPEAN PATENT APPLICATION**

21 Application number: 85112882.7

61 Int. Cl.<sup>4</sup>: B 41 J 3/04

22 Date of filing: 11.10.85

30 Priority: 15.10.84 US 661005

43 Date of publication of application:  
23.04.86 Bulletin 86/17

64 Designated Contracting States:  
BE DE FR GB IT NL

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54 Silicon nozzle structures and method of manufacture.

57 A nozzle structure in a crystallographically oriented, monocrystalline silicon includes a pyramidal opening (11) anisotropically etched from the entrance side of the nozzle and truncated in a membrane (12) having a smaller cross-section than the initial cross-section of the entrance opening. The membrane (12) has extending therethrough a pyramidal opening (13) etched anisotropically from the exit side. The vertical axes of both openings are substantially concentric.

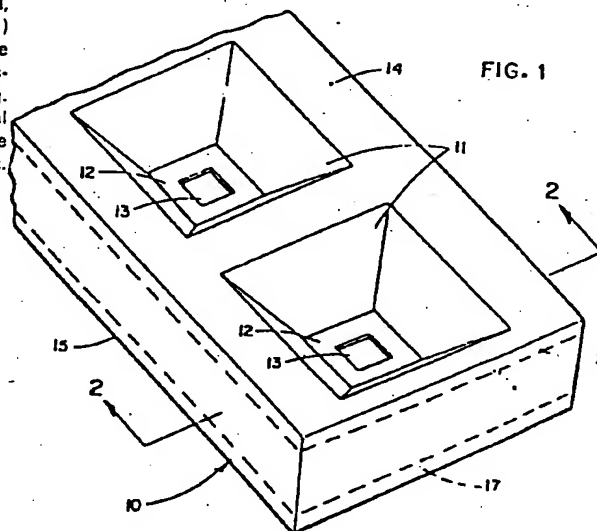


FIG. 1

## Silicon Nozzle Structures and Method of Manufacture

### Technical Field

Monocrystalline silicon bodies with passages.

### Background of the Invention

5 In the prior art and specifically in US-A-3,921,916 it is suggested that a monocrystalline, crystallographically oriented silicon wafer may be selectively etched to form one or more reproducible channels of a specific form in the wafer body. The specific type of the channel described in that patent has  
10 a rectangular entrance cross-section which continues to an intermediate rectangular cross-section, smaller than the entrance cross-section, and then to an exit cross-section which has a shape other than rectangular. A channel of this specific type is established by either  
15 of two disclosed processes, both of which utilize a heavily doped p+ layer (patterned in the one process and unpatterned in the other) as an etchant barrier. In the two processes, a silicon wafer is heavily doped to place it near or at saturation from one major face to form the  
20 p+ etchant barrier. Thereafter, patterned anisotropic etching from the opposite major face proceeds until the p+ barrier is reached. The anisotropic etching results in a rectangular entrance cross-section and a rectangular intermediate cross-section defining a membrane smaller  
25 in size than the entrance cross-section.

In the application of one process, the etching process is continued from the entrance side until an opening is made through the membrane. The other process utilizes patterned isotropic etching from the opposite

1 side (exit side) of the nozzle to complete a passage  
through the membrane to the intermediate cross-section.

Although these prior art processes may provide  
satisfactory ink jet nozzle structures, both of the  
5 described processes and the resulting structures have  
inherent problems. For example, due to inherent wafer  
thickness variations and isotropic etch nonuniformities,  
these processes require extensive mechanical and/or  
chemical polishing of both major surfaces of the wafer  
10 to improve dimensional control of the resulting nozzle  
structures. This is a costly processing step. Additionally,  
the nozzle structures produced by these processes have  
heavily saturated p+ regions surrounding the exit openings,  
and these regions tend to be brittle and thus subject to  
15 failure when exposed to high fluid pressures or pressure  
transients typically present in ink jet printing systems.

#### Disclosure of Invention

In accordance with the present invention, a standard  
commercially available semi-conductor wafer of crystallo-  
20 graphically oriented, monocrystalline p-type silicon is  
used to produce a single fluid nozzle or an array of  
nozzles directly and without the need for mechanical or  
chemical polishing of the two major surfaces of the wafer  
by a process wherein a low saturation n surface layer is  
25 formed on at least one major surface of the wafer. Materials  
resistant to an anisotropic etchant, later employed, are  
then deposited on both surfaces of the wafer. Thereafter,  
aperture masks defining the entrance and exit areas of  
a nozzle are formed on these major surfaces and the exit  
30 area is coated with a material which is both resistant  
to an etching solution and which provides an electrical  
connection to the n layer. A cavity is anisotropically  
etched from the entrance area of the wafer through to the  
n layer at the exit side by immersing the wafer in a  
35 caustic etching solution. A potential applied across the  
p/n junction at the exit side of the wafer electro-  
chemically stops the etching action leaving a membrane  
having a thickness substantially equal to the n-layer.

1 A passage is then anisotropically etched through the  
membrane from the exit side to complete the nozzle  
structure.

#### The Drawings

5 Fig. 1 shows a perspective view of a portion of  
the nozzle structure in accordance with the  
present invention;

Fig. 2 is a cross-sectional view of the nozzle  
structure taken along line 2-2 of Fig. 1;

10 Fig. 3 through 8 illustrate sequential cross-  
sectional views of a silicon wafer processed  
in accordance with the present invention.

#### Detailed Description

15 In multi-nozzle ink jet printing systems utilizing  
nozzles made of semi-conductor material, some of the more  
important characteristics required of the nozzle are the  
uniformity in the size of each respective nozzle, spatial  
distribution of the nozzles in an array, their  
20 resistance to cracking under the fluidic pressures  
encountered in the system, provision of an efficient  
mechanical impedance match between the fluid supply and  
the exit opening, as well as, their resistance to wear  
caused by the high velocity fluid flow through the  
nozzle structure.

25 Referring now to Fig. 1, there is shown a portion  
of the nozzle structure made in accordance with the  
present invention. Specifically a substrate 10 is shown  
having an array of uniform openings 11 therein. Each  
opening 11 starts with an initial, substantially square  
30 area and tapers to and terminates in a substantially  
square area smaller than the initial square area defining  
a membrane 12. As shown in Fig. 2, each membrane 12 in  
turn has an opening 13 extending therethrough which  
starts in a substantially square area smaller than the  
35 square area of each respective membrane 12 and terminates  
in a substantially square area larger than the starting  
square area of said opening. Both horizontal axes of the  
openings 13 in the membrane 12 are substantially aligned

1 with the horizontal axes of each corresponding opening 11  
in the main body of the wafer 10 by virtue of the  
wafer 10 crystallography.

Figs. 3 through 8 illustrate a sequence of  
5 process steps for production of an aperture in a single  
crystal silicon wafer 10 for forming one fluid nozzle or  
an array of nozzles. It is to be understood that the  
following process steps may be used in a different  
sequence and that other film materials for performing the  
10 same functions described below may be used. Furthermore,  
film formation, size, thickness and the like, may also be  
varied. The wafer 10 is of single crystal (100) oriented  
p type silicon with electrical resistivity of 0,5 to 100  
ohm-cm, approximately 19,5 to 20,5 mils thick having front  
15 14 and back 15 surfaces. The (100) planes are parallel  
to surfaces 14 and 15. As shown in Fig. 3, phosphorous  
is diffused into the front 14 and back 15 surfaces of the  
silicon wafer 10 to a depth of about 5 microns forming  
n type layers 16 and 17. As will become obvious later only  
20 one diffused layer is required to form a nozzle structure  
by the process (exit side). The diffusion is accomplished  
in a well-known manner by having a gas mixture containing  
0,75 %  $\text{PH}_3$ , 1 %  $\text{O}_2$ , and the make-up of Ar and  $\text{N}_2$  flow for  
30 minutes past the silicon wafer 10 which is maintained  
25 at 950°C. This is followed by a long drive-in period  
(1050°C for 22 hours) to achieve a thick layer (about 5  
microns). Since the final concentration of phosphorous  
in the n layers 16 and 17 is very low, this diffusion step  
introduces very little stress into the silicon wafer 10,  
30 and consequently the silicon structure retains its  
strength.

Next as shown in Fig. 4, both front 14 and back  
15 surfaces of the wafer 10 are coated with a protective  
material such as LPCVD silicon nitride forming layers 18  
35 and 19 which can resist a long etching period in a  
caustic (KOH) solution. One of the ways to accomplish this  
is to utilize a low pressure chemical vapor deposition  
of silicon nitride deposited at about 800°C. Oxide layers

1 (not shown) less than 0,5 microns thick may be grown on  
both sides of layers 18 and 19 to reduce the effect of  
stress between nitride and silicon and to improve adhesion  
of photoresist to nitride. To promote ease of photoshaping  
5 it is recommended that the wafer 10 when procured have  
its back surface 15 etched in an acidic rather than  
caustic solution.

Thereafter, masks are prepared corresponding  
to the desired entrance 20 and exit 21 areas of the nozzle.  
10 The masks for both entrance 20 and exit 21 areas are made  
circular in shape since the openings in the silicon wafer  
10 defined by circular masks will etch out to squares  
parallel to the 100 planes, each square circumscribing  
its respective circle. Use of circular masks eliminates  
15 possible error due to the theta misalignment which may  
occur when a square shaped mask is used. The silicon  
nitride layers 18 and 19 are photoshaped simultaneously  
on both sides using a two-sided photospinner (not shown)  
and a two-sided aligner (not shown). The resulting  
20 structure after etching away of portions of layers 18 and  
19 defining the entrance 20 and exit 21 areas, is shown  
in Fig. 5.

The exit area 21 is then protected from the  
etching solution by covering it with a metallic layer 22,  
25 as shown in Fig. 6, or by use of a hermetic mechanical  
fixture (not shown). Thereafter the wafer is submerged  
in a hot (80-85°C) KOH solution (not shown) and a potential  
is placed across the p/n junction at the back side 15 by  
connecting the positive side of an electrical power source  
30 (not shown) with the metallic layer 22 protecting the exit  
area 21. Other alkaline etch solutions such as metal  
hydroxides of the Group I-A elements of the Periodic Table,  
for example, NaOH,  $\text{NH}_4\text{OH}$ , or others, may be used. The use  
of electrochemically controlled thinning process for  
35 semi-conductors is well-known in the art and is described  
in detail in US-A-3,689,389.

The opening 11 in the monocrystalline silicon  
wafer 10 is etched anisotropically until the diffused

1 layer 17 at the back side 25 is reached, at which time  
the etching action stops due to an oxide layer (not shown)  
which is caused to grow at the p/n junction due to the  
5 applied potential across the junction. It is well known  
in the art that the (111) plane is a slow etch plane in  
monocrystalline silicon material when a KOH etching  
solution is used. Thus, the etching step produces a  
pyramidal opening in the wafer 10 which opening truncates  
10 in a membrane 12 when it encounters the electrochemical  
etch barrier set up at the silicon and diffused layer 17  
interface (p/n junction).

Thereafter, the wafer 10 is removed from the  
etching solution, the protective metallic layer 22 and  
associated electrical connection on the exit side are  
15 removed, and the entrance side 20 is protected from the  
etching solution usually by a layer 24 formed by air  
oxidation. The wafer 10 is then re-submersed into the  
etching solution and a pyramidal passage is etched aniso-  
tropically from the back surface 15 to form the exit  
20 opening 13. The resulting structure is shown in Fig. 7.

If desired, the protective coatings 18, 19 and  
24 are then removed leaving a completed pure silicon  
nozzle structure as shown in Fig. 8. Typically the initial  
opening of the entrance 20 is about 35 mils wide and the  
25 smallest portion of the exit opening 13 is about 1.5 to  
4 mils wide.

Since the etch rate perpendicular to the (111)  
planes is very low compared to the vertical etch rate (100),  
overetch does not mitigate against the high accuracy  
30 defined by the exit mask. To prevent ink from wetting the  
surface of the wafer on the exit side, the back surface  
15 of the wafer 10 may be coated with a material of low  
surface energy such as Teflon.

Claims

1. A nozzle comprising:  
a nozzle body (10) formed of a semiconductor material  
having a rectangular entrance aperture (11) of a first  
cross-sectional area which tapers to a second rectangular  
cross-sectional area which is smaller than the first cross-  
sectional area of said entrance aperture; and  
a membrane (12) of said semiconductor material formed  
within said second cross-sectional area, said membrane  
(12) having a rectangular exit aperture (13) therein, said  
exit aperture (13) having a first cross-sectional area  
which is smaller than the second cross-sectional area of  
the entrance aperture (11), said first cross-sectional  
area of the exit aperture (13) tapering to a second cross-  
sectional area which is larger than said first cross-  
sectional area of said exit aperture (13).
2. The nozzle in accordance with claim 1  
wherein said semiconductor material is monocrystalline  
silicon.
3. The nozzle in accordance with claim 2  
wherein said entrance (11) and exit (12) apertures have  
substantially square cross-sections.
4. The nozzle in accordance with claim 3  
wherein said cross-sections are substantially parallel  
to the (100) planes of the monocrystalline silicon.
5. The nozzle in accordance with claim 3  
wherein said entrance (11) and exit (13) apertures are  
substantially concentric.
6. The nozzle in accordance with claim 5  
wherein the thickness of said membrane (12) is 10 microns  
or less.
7. The nozzle in accordance with claim 6  
wherein said membrane (12) is of n type silicon.



1           8.     A process for forming a nozzle structure  
comprising an aperture in a section (10) of crystallo-  
graphically oriented, p-type, monocrystalline silicon  
having first (15) and second (14) major surfaces, the  
5 process comprising:  
forming a n surface layer (17) on the first major surface  
(15) of the silicon section (10);  
anisotropically etching a cavity from the second surface  
(14) of the silicon section to said n layer (17); and  
10 anisotropically etching a passage (13) through said n  
layer (17) from the first surface (15) of the silicon  
section (10).

          9.     The process of claim 8  
wherein said step of anisotropically etching a cavity  
15 includes establishing an electrochemical barrier at the  
silicon n layer interface to stop the etching process.

          10.    The process of claim 9  
wherein said step of anisotropically etching said cavity  
further includes:  
20 coating said first and second surfaces (14, 15) of the  
silicon section (10) with a material (18, 19) which  
resists etching;  
removing said coating from said first and second surfaces  
in the area (20, 21) defining the entrance (11) and exit  
25 (13) of the nozzle respectively;  
protecting the exit area (21) from the etching solution  
with a means (22) which is an electrical conductor;  
immersing said silicon section (10) in an etching solution;  
immersing a cathode into the etching solution;  
30 applying a positive potential to said protecting means  
(22) thereby establishing a potential across the p/n  
junction formed by the silicon (10) and the n layer (17).

          11.    The process of claim 10  
wherein said step of coating said first and second surfaces  
35 (14, 15) comprises:  
growing an oxide layer on said first and second surfaces  
(14, 15);

- 1 depositing silicon nitride (18, 19) on said oxidized surfaces; and  
growing an oxide layer on said nitride layers (18, 19).
12. The process of claim 11
- 5 wherein the process of removing said coating comprises:  
coating said first and second surfaces of the silicon with photoresist;  
exposing the photoresist on both sides of the silicon to define the entrance (20) and exit (21) areas of the  
10 nozzle;  
etching away the oxide-nitride-oxide layers (18, 19) from the areas (20, 21) defining the entrance (11) and exit (13) of the nozzle.
13. The process of claim 12
- 15 wherein said exposed exit (13) and entrance (11) areas have circular shapes.
14. The process of claim 10 wherein said etching solution is a KOH solution.
15. The process of claim 8
- 20 wherein said cavity and passage are concentric.
16. A process for forming a nozzle structure comprising an aperture in a thin section of crystallographically oriented, p-type, monocrystalline silicon having front and back plane surfaces (14, 15), the  
25 process comprising:  
forming an n surface layer (17) on the back surface (15) of the silicon section (10);  
coating both front and back surfaces (14, 15) of the silicon section (10) except for the areas (20, 21) which  
30 define the entrance (11) and the exit (13) of the nozzle with a material (18, 19) which resists etching;  
applying a protective coating (22) to the exit side of the aperture;  
immersing the silicon section (10) in an etching solution;  
35 immersing in said solution a cathode;  
applying a controlling positive potential to the n layer (17);

- 1 anisotropically etching a cavity from the entrance side  
of the nozzle through to the n layer (17) at the exit side  
of the nozzle;  
removing the silicon section (10) from the etching solution;
- 5 removing the protecting layer (22) covering the exit area  
of the nozzle;  
coating the entrance side with a protective layer (24);  
re-immersing the silicon body (10) in the etching  
solution; and
- 10 anisotropically etching a passage (13) from the back  
surface of the silicon section (10) through the n layer  
(17) to the cavity.

17. The process of claim 16  
wherein said monocrystalline silicon is oriented along  
15 the (100) or (110) planes.

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FIG. 1

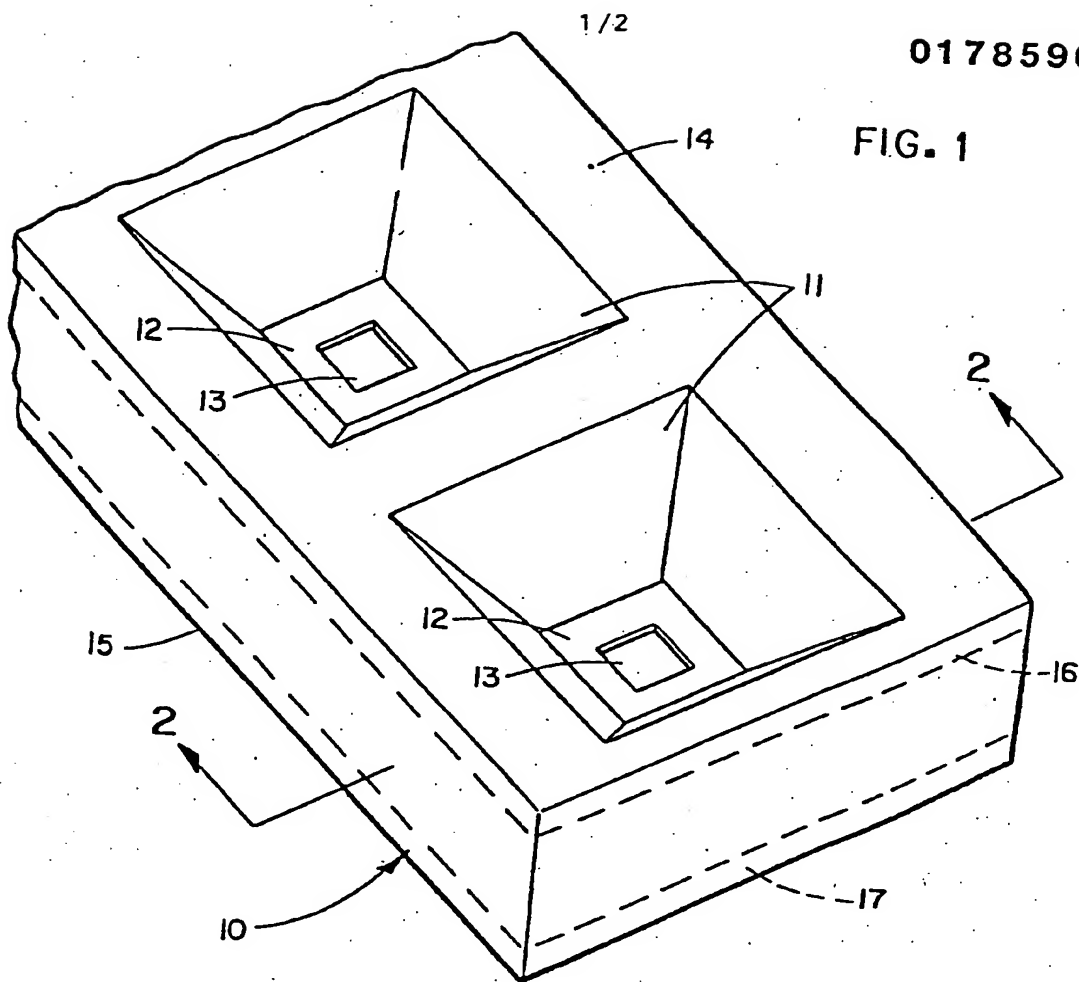


FIG. 2

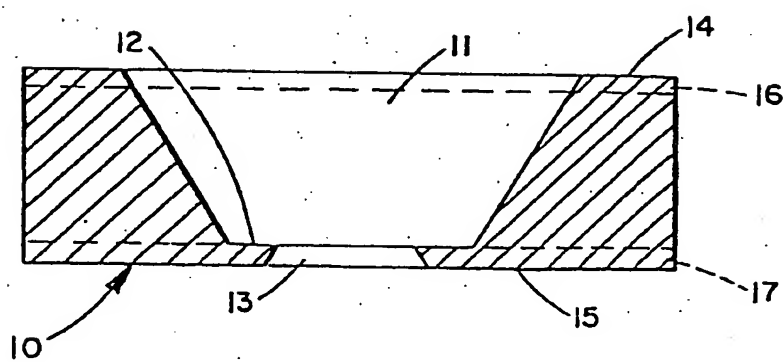


FIG. 3

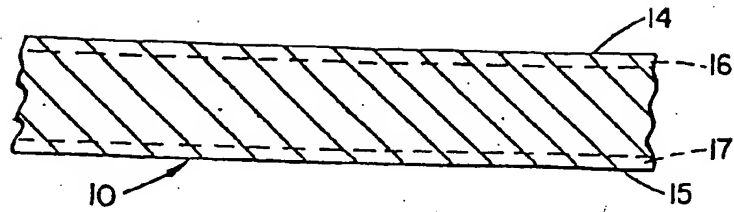


FIG. 4

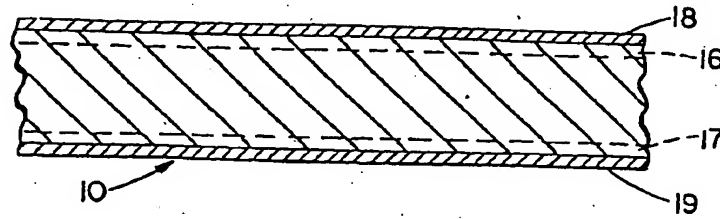


FIG. 5

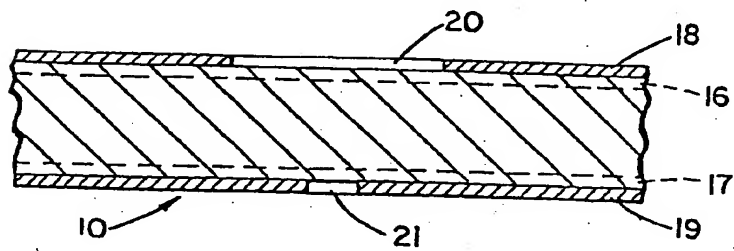


FIG. 6

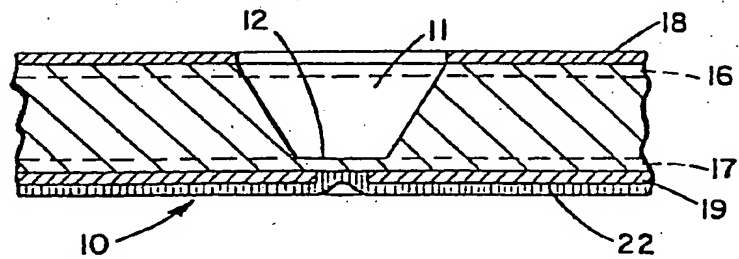


FIG. 7

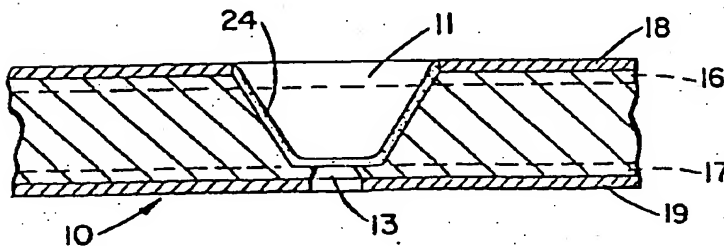
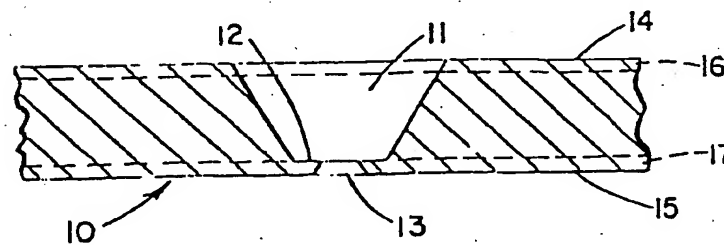


FIG. 8



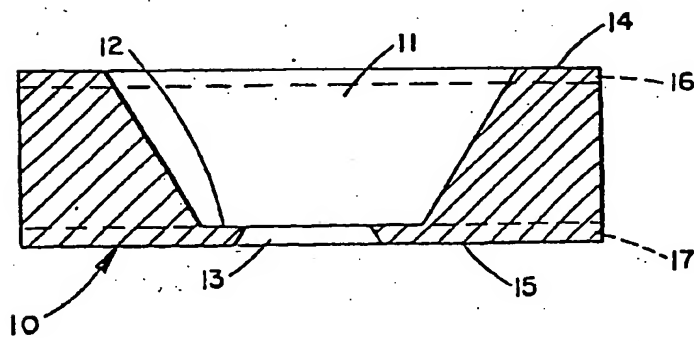
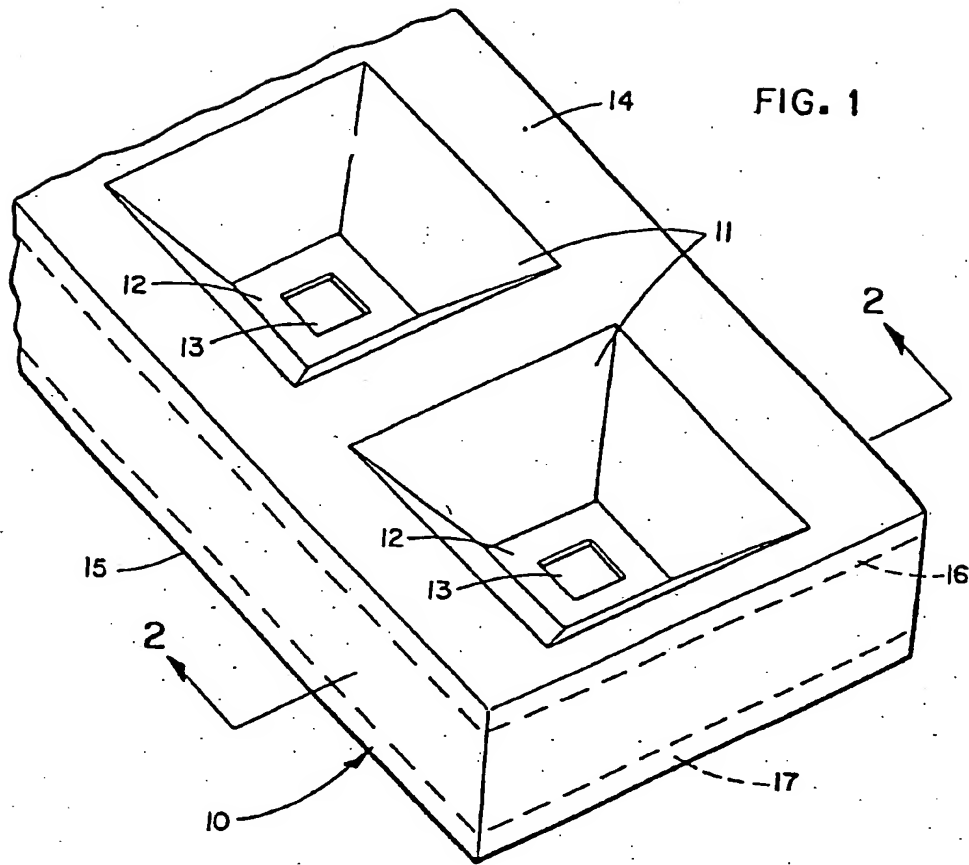


FIG. 3

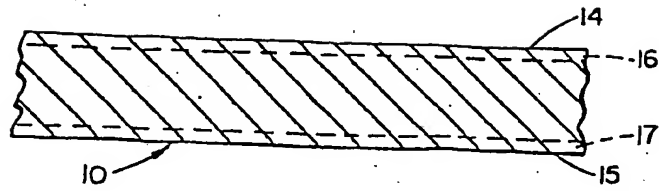


FIG. 4

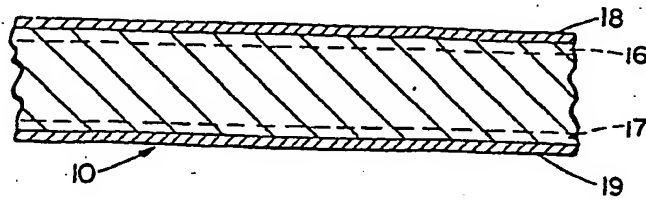


FIG. 5

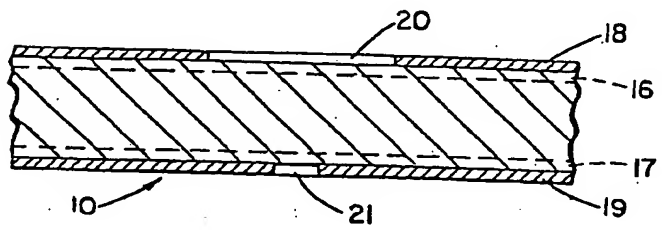


FIG. 6

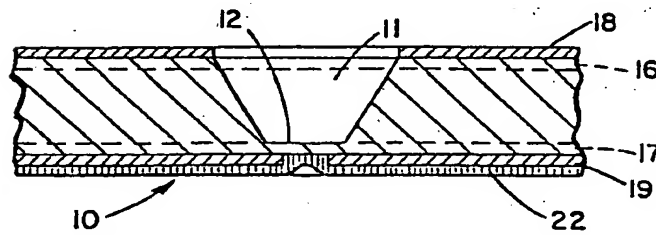


FIG. 7

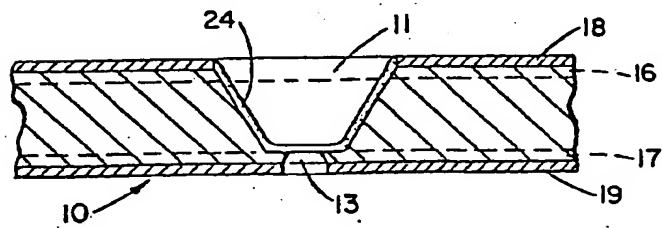
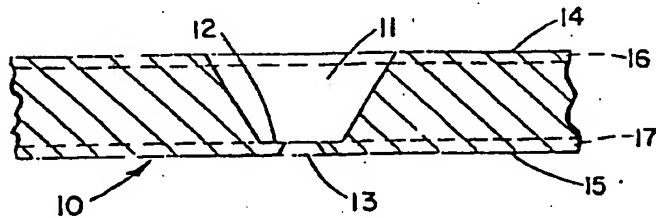


FIG. 8



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**EUROPEAN PATENT SPECIFICATION**

⑬ Date of publication of patent specification: 16.01.91

⑭ Int. Cl.<sup>5</sup>: **B 41 J 2/135, B 41 J 2/16,**  
**B 05 B 1/00**

⑮ Application number: 85112882.7

⑯ Date of filing: 11.10.85

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⑱ Priority: 15.10.84 US 661005

⑲ Date of publication of application:  
23.04.86 Bulletin 86/17

⑳ Publication of the grant of the patent:  
16.01.91 Bulletin 91/03

㉑ Designated Contracting States:  
BE DE FR GB IT NL

㉒ References cited:  
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US-A-3 921 916  
US-A-4 157 935  
US-A-4 455 192

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Courier Press, Leamington Spa, England.

**EP 0 178 596 B1**



## Description

### Technical field

Monocrystalline silicon bodies with passages.

### Background of the invention

In the prior art and specifically in US-A-3,921,916 it is suggested that a monocrystalline, crystallographically oriented silicon wafer may be selectively etched to form one or more reproducible channels of a specific form in the wafer body. The specific type of the channel described in that patent has a rectangular entrance cross-section which continues to an intermediate rectangular cross-section smaller than the entrance cross-section, and then to an exit cross-section which has a shape other than rectangular. A channel of this specific type is established by either of two disclosed processes, both of which utilize a heavily doped p+ layer (patterned in the one process and unpatterned in the other) as an etchant barrier. In the two processes, a silicon wafer is heavily doped to place it near or at saturation from one major face to form the p+ etchant barrier. Thereafter, patterned anisotropic etching from the opposite major face proceeds until the p+ barrier is reached. The anisotropic etching results in a rectangular entrance cross-section and a rectangular intermediate cross-section defining a membrane smaller in size than the entrance cross-section.

In the application of one process, the etching process is continued from the entrance side until an opening is made through the membrane. The other process utilizes patterned isotropic etching from the opposite side (exit side) of the nozzle to complete a passage through the membrane to the intermediate cross-section.

Although these prior art processes may provide satisfactory ink jet nozzle structures, both of the described processes and the resulting structures have inherent problems. For example, due to inherent wafer thickness variations and isotropic etch nonuniformities, these processes require extensive mechanical and/or chemical polishing of both major surfaces of the wafer to improve dimensional control of the resulting nozzle structures. This is a costly processing step. Additionally, the nozzle structures produced by these processes have heavily saturated p+ regions surrounding the exit openings, and these regions tend to be brittle and thus subject to failure when exposed to high fluid pressures or pressure transients typically present in ink jet printing systems.

### Disclosure of invention

In accordance with the present invention, a standard commercially available semi-conductor wafer of crystallographically oriented, monocrystalline p-type silicon is used to produce a single fluid nozzle or an array of nozzles directly and without the need for mechanical or chemical polishing of the two major surfaces of the wafer

by a process wherein a low saturation n surface layer is formed on at least one major surface of the wafer. Materials resistant to an anisotropic etchant, later employed, are then deposited on both surfaces of the wafer. Thereafter, aperture masks defining the entrance and exit areas of a nozzle are formed on these major surfaces and the exit area is coated with a material which is both resistant to an etching solution and which provides an electrical connection to the n layer. A cavity is anisotropically etched from the entrance area of the wafer through to the n layer at the exit side by immersing the wafer in a caustic etching solution. A potential applied across the p/n junction at the exit side of the wafer electrochemically stops the etching action leaving a membrane having a thickness substantially equal to the n-layer. A passage is then anisotropically etched through the membrane from the exit side to complete the nozzle structure.

### The drawings

Fig. 1 shows a perspective view of a portion of the nozzle structure in accordance with the present invention;

Fig. 2 is a cross-sectional view of the nozzle structure taken along line 2—2 of Fig. 1;

Fig. 3 through 8 illustrates sequential cross-sectional views of a silicon wafer processed in accordance with the present invention.

### Detailed description

In multi-nozzle ink jet printing systems utilizing nozzles made of semi-conductor material, some of the more important characteristics required of the nozzle are the uniformity in the size of each respective nozzle, spatial distribution of the nozzles in an array, their resistance to cracking under the fluidic pressures encountered in the system, provision of an efficient mechanical impedance match between the fluid supply and the exit opening, as well as, their resistance to wear caused by the high velocity fluid flow through the nozzle structure.

Referring now to Fig. 1, there is shown a portion of the nozzle structure made in accordance with the present invention. Specifically a substrate 10 is shown having an array of uniform openings 11 therein. Each opening 11 starts with an initial, substantially square area and tapers to and terminates in a substantially square area smaller than the initial square area defining a membrane 12. As shown in Fig. 2, each membrane 12 in turn has an opening 13 extending therethrough which starts in a substantially square area smaller than the square area of each respective membrane 12 and terminates in a substantially square area larger than the starting square area of said opening. Both horizontal axes of the openings 13 in the membrane 12 are substantially aligned with the horizontal axes of each corresponding opening 11 in the main body of the wafer 10 by virtue of the wafer 10 crystallography.

Figs. 3 through 8 illustrate a sequence of process steps for production of an aperture in a

single crystal silicon wafer 10 for forming one fluid nozzle or an array of nozzles. It is to be understood that the following process steps may be used in a different sequence and that other film materials for performing the same functions described below may be used. Furthermore, film formation, size, thickness and the like, may also be varied. The wafer 10 is of single crystal (100) oriented p-type silicon with electrical resistivity of 0.5 to 100 ohm-cm, approximately 495  $\mu\text{m}$  to 515  $\mu\text{m}$  (19.5 to 20.5 mils) thick having front 14 and back 15 surfaces. The (100) planes are parallel to surfaces 14 and 15. As shown in Fig. 3, phosphorous is diffused into the front 14 and back 15 surfaces of the silicon wafer 10 to a depth of about 5  $\mu\text{m}$  forming n-type layers 16 and 17. As will become obvious later only one diffused layer is required to form a nozzle structure by the process (exit side). The diffusion is accomplished in a well-known manner by having a gas mixture containing 0.75%  $\text{PH}_3$ , 1%  $\text{O}_2$ , and the make-up of Ar and  $\text{N}_2$  flow for 30 minutes past the silicon wafer 10 which is maintained at 950°C. This is followed by a long drive-in period (1050°C for 22 hours) to achieve a thick layer (about 5 microns). Since the final concentration of phosphorous in the n layers 16 and 17 is very low, this diffusion step introduces very little stress into the silicon wafer 10, and consequently the silicon structure retains its strength.

Next, as shown in Fig. 4, both front 14 and back 15 surfaces of the wafer 10 are coated with a protective material such as LPCVD silicon nitride forming layers 18 and 19 which can resist a long etching period in a caustic (KOH) solution. One of the ways to accomplish this is to utilize a low pressure chemical vapor deposition of silicon nitride deposited at about 800°C. Oxide layers (not shown) less than 0.5  $\mu\text{m}$  thick may be grown on both sides of layers 18 and 19 to reduce the effect of stress between nitride and silicon and to improve adhesion of photoresist to nitride. To promote ease of photoshaping it is recommended that the wafer 10 when procured have its back surface 15 etched in an acidic rather than caustic solution.

Thereafter, masks are prepared corresponding to the desired entrance 20 and exit 21 areas of the nozzle. The masks for both entrance 20 and exit 21 areas are made circular in shape since the openings in the silicon wafer 10 defined by circular masks will etch out to squares parallel to the 100 planes, each square circumscribing its respective circle. Use of circular masks eliminates possible error due to the theta misalignment which may occur when a square shaped mask is used. The silicon nitride layers 18 and 19 are photoshaped simultaneously on both sides using a two-sided photospinner (not shown) and a two-sided aligner (not shown). The resulting structure after etching away of portions of layers 18 and 19 defining the entrance 20 and exit 21 areas, is shown in Fig. 5.

The exit area 21 is then protected from the etching solution by covering it with a metallic layer 22, as shown in Fig. 6, or by use of a

hermetic mechanical fixture (not shown). Thereafter the wafer is submerged in a hot (80—85°C) KOH solution (not shown) and a potential is placed across the p/n junction at the back side 15 by connecting the positive side of an electrical power source (not shown) with the metallic layer 22 protecting the exit area 21. Other alkaline etch solutions such as metal hydroxides of the Group I—A elements of the Periodic Table, for example,  $\text{NaOH}$ ,  $\text{NH}_4\text{OH}$ , or others, may be used. The use of electrochemically controlled thinning process for semi-conductors is well-known in the art and is described in detail in US—A—3,689,389.

The opening 11 in the monocrystalline silicon wafer 10 is etched anisotropically until the diffused layer 17 at the back side 25 is reached, at which time the etching action stops due to an oxide layer (not shown) which is caused to grow at the p/n junction due to the applied potential across the junction. It is well-known in the art that the (111) plane is a slow etch plane in monocrystalline silicon material when a KOH etching solution is used. Thus, the etching step produces a pyramidal opening in the wafer 10 which opening truncates in a membrane 12 when it encounters the electrochemical etch barrier set up at the silicon and diffused layer 17 interface (p/n junction).

Thereafter, the wafer 10 is removed from the etching solution, the protective metallic layer 22 and associated electrical connection on the exit side are removed, and the entrance side 20 is protected from the etching solution usually by a layer 24 formed by air oxidation. The wafer 10 is then re-submerged into the etching solution and a pyramidal passage is etched anisotropically from the back surface 15 to form the exit opening 13. The resulting structure is shown in Fig. 7.

If desired, the protective coatings 18, 19 and 24 are then removed leaving a completed pure silicon nozzle structure as shown in Fig. 8. Typically the initial opening of the entrance 20 is about 890  $\mu\text{m}$  (35 mils) wide and the smallest portion of the exit opening 13 is about 38  $\mu\text{m}$  to 102  $\mu\text{m}$  (1.5 to 4 mils) wide.

Since the etch rate perpendicular to the (111) planes is very low compared to the vertical etch rate (100), overetch does not mitigate against the high accuracy defined by the exit mask. To prevent ink from wetting the surface of the wafer on the exit side, the back surface 15 of the wafer 10 may be coated with a material of low surface energy such as Teflon.

## Claims

1. A nozzle comprising a nozzle body (10) formed of a monocrystalline p-type silicon material having a rectangular entrance aperture (11) of a first cross-sectional area which tapers to a second rectangular cross-sectional area which is smaller than the first cross-sectional area of said entrance aperture; and a membrane (12) of said semi-conductor material formed within said second cross-sectional area.

tional area and having an exit aperture (13) characterized in that said membrane (12) is of n-type silicon and has a rectangular exit aperture (13) therein, said exit aperture (13) having a first cross-sectional area which is smaller than the second cross-sectional area of the entrance aperture (11), said first cross-sectional area of the exit aperture (13) tapering to a second cross-sectional area which is larger than said first cross-sectional area of said exit aperture (13).

2. The nozzle in accordance with claim 1 wherein said entrance (11) and exit (12) apertures have substantially square cross-sections.

3. The nozzle in accordance with claim 2 wherein said cross-sections are substantially parallel to the (100) planes of the monocrystalline silicon.

4. The nozzle in accordance with claim 2 wherein said entrance (11) and exit (13) apertures are substantially concentric.

5. The nozzle in accordance with claim 4 wherein the thickness of said membrane (12) is 10 micrometers or less.

6. A process for forming a nozzle structure comprising an aperture in a section (10) of crystallographically oriented, p-type, monocrystalline silicon having first (15) and second (14) major surfaces, the process comprising:

forming an n surface layer (17) on the first major surface (15) of the silicon section (10);

anisotropically etching a cavity from the second surface (14) of the silicon section to said n layer (17); and

anisotropically etching a passage (13) through said n layer (17) from the first surface (15) of the silicon section (10).

7. The process of claim 6 wherein said step of anisotropically etching a cavity includes establishing an electrochemical barrier at the silicon n layer interface to stop the etching process.

8. The process of claim 7 wherein said step of anisotropically etching said cavity further includes:

coating said first and second surfaces (14, 15) of the silicon section (10) with a material (18, 19) which resists etching;

removing said coating from said first and second surfaces in the area (20, 21) defining the entrance (11) and exit (13) of the nozzle respectively;

protecting the exit area (21) from the etching solution with a means (22) which is an electrical conductor;

immersing said silicon section (10) in an etching solution;

immersing a cathode into the etching solution; applying a positive potential to said protecting means (22) thereby establishing a potential across the p/n junction formed by the silicon (10) and the n layer (17).

9. The process of claim 8 wherein said step of coating said first and second surfaces (14, 15) comprises:

growing an oxide layer on said first and second surfaces (14, 15);

depositing silicon nitride (18, 19) on said oxidized surfaces; and

growing an oxide layer on said nitride layers (18, 19).

10. The process of claim 9 wherein the process of removing said coating comprises:

coating said first and second surfaces of the silicon with photoresist;

exposing the photoresist on both sides of the silicon to define the entrance (20) and exit (21) areas of the nozzle;

etching away the oxide-nitride-oxide layers (18, 19) from the areas (20, 21) defining the entrance (11) and exit (13) of the nozzle.

11. The process of claim 10 wherein said exposed exit (13) and entrance (11) areas have circular shapes.

12. The process of claim 8 wherein said etching solution is a KOH solution.

13. The process of claim 6 wherein said cavity and passage are concentric.

14. A process for forming a nozzle structure comprising an aperture in a thin section of crystallographically oriented, p-type, monocrystalline silicon having front and back plane surfaces (14, 15), the process comprising:

forming an n surface layer (17) on the back surface (15) of the silicon section (10);

coating both front and back surfaces (14, 15) of the silicon section (10) except for the areas (20, 21) which define the entrance (11) and the exit (13) of the nozzle with a material (18, 19) which resists etching;

applying a protective coating (22) to the exit side of the aperture;

immersing the silicon section (10) in an etching solution;

immersing in said solution a cathode;

applying a controlling positive potential to the n layer (17);

anisotropically etching a cavity from the entrance side of the nozzle through to the n layer (17) at the exit side of the nozzle;

removing the silicon section (10) from the etching solution;

removing the protecting layer (22) covering the exit area of the nozzle;

coating the entrance side with a protective layer (24);

re-immersing the silicon body (10) in the etching solution; and

anisotropically etching a passage (13) from the back surface of the silicon section (10) through the n layer (17) to the cavity.

15. The process of claim 14 wherein said monocrystalline silicon is oriented along the (100) of (110) planes.

#### Patentansprüche

1. Düse mit einem Düsenkörper (10), der aus einem monokristallinen p-Siliziummaterial geformt ist und der eine Eingangsöffnung (11) aufweist mit einer ersten rechteckigen Grundfläche, die konisch zuläuft zu einer zweiten rechtecki-

gen Grundfläche, die kleiner ist als die erste Grundfläche, und

einer Membran (12) aus dem vorgenannten Halbleitermaterial, die in der zweiten Grundfläche erzeugt ist und eine Ausgangsöffnung (13) aufweist, dadurch gekennzeichnet,

daß die Membran (12) aus n-Silicium besteht und eine rechteckige Ausgangsöffnung (13) besitzt,

daß die Ausgangsöffnung (13) eine erste Grundfläche aufweist, die kleiner ist als die zweite Grundfläche der Eingangsöffnung (11) und

daß die erste Grundfläche der Ausgangsöffnung (13) konisch zuläuft zu einer zweiten Grundfläche, die größer ist als die erste Grundfläche der Ausgangsöffnung (13).

2. Düse nach Anspruch 1, dadurch gekennzeichnet, daß deren Eingangs- und Ausgangsöffnung (11, 13) im wesentlichen quadratisch sind.

3. Düse nach Anspruch 2, dadurch gekennzeichnet, daß die Grundflächen im wesentlichen parallel zu den (100)-Ebenen des monokristallinen Siliciums verlaufen.

4. Düse nach Anspruch 2, dadurch gekennzeichnet, daß die Eingangs- und Ausgangsöffnung (11, 13) im wesentlichen konzentrisch sind.

5. Düse nach Anspruch 4, dadurch gekennzeichnet, daß die Membrandicke (12) 10 µm oder weniger beträgt.

6. Verfahren zur Herstellung einer Düsenstruktur, die eine Öffnung in einem Segment (10) aus kristallographisch orientiertem, monokristallinem p-Silicium enthält, mit einer ersten (15) und einer zweiten (14) Hauptfläche, mit den Verfahrensschritten:

Herstellen einer n-Oberflächenschicht (17) auf der ersten Hauptfläche (15) des Siliciumsegments (10);

anisotropes Ätzen eines Hohlraumes (11), ausgehend von der zweiten Hauptfläche (14) des Siliciumsegment bis zu der n-Schicht (17); und

anisotropes Ätzen einer Öffnung (13) durch die n-Schicht (17) von der ersten Hauptfläche (15) des Siliciumsegments (10) aus.

7. Verfahren nach Anspruch 6, dadurch gekennzeichnet, daß der Schritt des anisotropen Ätzens eines Hohlraumes einschließt:

Aufbringen einer elektrochemischen Sperre an der Grenzfläche zwischen Silicium und n-Schicht, um den Ätzprozeß zu stoppen.

8. Verfahren nach Anspruch 7, dadurch gekennzeichnet, daß der Schritt des anisotropen Ätzens des Hohlraumes weiterhin einschließt:

Beschichten der ersten und zweiten Hauptfläche (14, 15) des Siliciumsegments (10) mit einem gegen Ätzen beständigen Material (18, 19);

Entfernen der Beschichtung von der ersten und zweiten Hauptfläche in den Bereichen (20, 21), die den Eingang (11) bzw. Ausgang (13) der Düse definieren;

Schützen des Ausgangsbereiches (21) vor der Ätzlösung mit einem elektrisch leitenden Mittel (22);

Eintauchen des Siliciumsegments (10) in eine Ätzlösung;

Eintauchen einer Kathode in die Ätzlösung;  
Anlegen eines positiven Potentials an das Schutzmittel (22), um hierdurch ein Potential entlang des pn-Übergangs, der durch das Silicium (10) und die n-Schicht (17) gebildet wird, zu erzeugen.

9. Verfahren nach Anspruch 8, dadurch gekennzeichnet, daß der Schritt des Beschichtens der ersten und zweiten Hauptfläche (14, 15) enthält:

Aufwachsen einer Oxidschicht auf die erste und zweite Hauptfläche (14, 15);

Abscheiden von Siliciumnitrid (18, 19) auf den oxidierten Hauptflächen; und

Aufwachsen einer Oxidschicht auf den Nitridschichten (18, 19).

10. Verfahren nach Anspruch 9, dadurch gekennzeichnet, daß das Verfahren zum Entfernen der Beschichtung umfaßt:

Beschichten der ersten und zweiten Hauptfläche des Siliciums mit einem Photoresist;

Belichten des Photoresist auf beiden Seiten des Siliciums, um den Eingangs- (20) und den Ausgangsbereich (21) der Düse zu definieren;

Wegätzen der Oxid-Nitrid-Oxid-Schichten (18, 19) von den Bereichen (20, 21), die den Eingang (11) und Ausgang (13) der Düse definieren.

11. Verfahren nach Anspruch 10, dadurch gekennzeichnet, daß die belichteten Ausgangs- (13) und Eingangsflächen (11) einen kreisförmigen Umriß aufweisen.

12. Verfahren nach Anspruch 8, dadurch gekennzeichnet, daß die Ätzlösung eine KOH-Lösung ist.

13. Verfahren nach Anspruch 6, dadurch gekennzeichnet, daß der Hohlraum und die Öffnung konzentrisch sind.

14. Verfahren zur Herstellung einer Düsenstruktur, bestehend aus einer Öffnung in einem dünnen Segment aus kristallographisch orientiertem, monokristallinem p-Silicium, das eine ebene Vorder- und Rückfläche (14, 15) aufweist, mit den Verfahrensschritten:

Bilden einer n-Oberflächenschicht (17) auf der Rückfläche (15) des Siliciumsegmentes;

Beschichten der Vorder- und Rückfläche (14, 15) des Siliciumsegmentes (10) mit Ausnahme der Bereiche, die den Eingang (11) und Ausgang (13) der Düse definieren, mit einem Material (18, 19), das gegen Ätzen beständig ist;

Aufbringen einer Schutzschicht (22) auf die Ausgangsseite der Öffnung;

Eintauchen des Siliciumsegmentes (10) in eine Ätzlösung;

Eintauchen einer Kathode in die Ätzlösung;  
Anlegen eines kontrollierten positiven Potentials an die n-Schicht (17);

anisotropes Ätzen eines Hohlraumes von der Eingangsseite der Düse bis zur n-Schicht (17) an der Ausgangsseite der Düse;

Herausnehmen des Siliciumsegmentes (10) aus der Ätzlösung;

Entfernen der Schutzschicht (22), die die Ausgangsfläche der Düse bedeckt;

Beschichten der Eingangsseite mit einer Schutzschicht (24);

Wiedereintauchen des Siliciumkörpers (10) in die Ätzlösung; und

anisotropes Ätzen einer Öffnung (13) von der Rückfläche des Siliciumsegmentes (10) durch die n-Schicht (17) bis zu dem Hohlraum.

15. Verfahren nach Anspruch 14, dadurch gekennzeichnet, daß das monokristalline Silicium entlang der (100)- oder (110)-Ebene orientiert ist.

#### Revendications

1. Buse comprenant un corps (10) de buse formé d'une matière de type silicium monocristallin de type p présentant une ouverture d'entrée rectangulaire (11) d'une première coupe transversale qui s'effile jusqu'à une seconde coupe transversale rectangulaire qui est plus petite que la première coupe transversale de ladite ouverture d'entrée; et

une membrane (12) de ladite matière semi-conductrice formée à l'intérieur de ladite seconde coupe transversale et ayant une ouverture de sortie (13), caractérisée en ce que ladite membrane (12) est en silicium de type n et présente une ouverture rectangulaire (13) de sortie, ladite ouverture (13), de sortie ayant une première coupe transversale qui est plus petite que la seconde coupe transversale de l'ouverture d'entrée (11), ladite première coupe transversale de l'ouverture de sortie (13) s'amincissant vers une seconde coupe transversale qui est plus grande que ladite première coupe transversale de ladite ouverture de sortie (13).

2. Buse selon la revendication 1, dans laquelle lesdites ouvertures d'entrée (11) et de sortie (13) ont des coupes transversales sensiblement carrées.

3. Buse selon la revendication 2, dans laquelle lesdites coupes transversales sont sensiblement parallèles aux plans (100) du silicium monocristallin.

4. Buse selon la revendication 2, dans laquelle lesdites ouvertures d'entrée (11) et de sortie (13) sont sensiblement concentriques.

5. Buse selon la revendication 4, dans laquelle l'épaisseur de ladite membrane (12) est de 10 micromètres ou moins.

6. Procédé pour former une structure de buse comprenant une ouverture dans une partie (10) de silicium monocristallin de type p, à orientation cristallographique, présentant des première (15) et seconde (14) surfaces principales, le procédé consistant:

à former une couche superficielle n (17) sur la première surface principale (15) de la partie (10) en silicium;

à graver de façon anisotrope une cavité depuis la seconde surface (14) de la partie en silicium jusqu'à ladite couche n (17); et

à graver de façon anisotrope un passage (13) à travers ladite couche n (17) depuis la première surface (15) de la partie (10) en silicium.

7. Procédé selon la revendication 6, dans lequel ladite étape de gravure anisotrope d'une cavité consiste à établir une couche d'arrêt électrochimique

que à l'interface du silicium et de la couche n pour arrêter le processus de gravure.

8. Procédé selon la revendication 7, dans lequel ladite étape de gravure anisotrope de ladite cavité consiste en outre:

à revêtir lesdites première et secondes surfaces (14, 15) de la section (10) en silicium d'une matière (18, 19) qui résiste à la gravure;

à enlever ledit revêtement desdites première et seconde surfaces dans la section (20, 21) définissant l'entrée (11) et la sortie (13) de la buse, respectivement;

à protéger la section de sortie (21) de la solution de gravure à l'aide d'un moyen (22) qui est un conducteur électrique;

à immerger ladite tranche (10) en silicium dans une solution de gravure;

à immerger une cathode dans la solution de gravure;

à appliquer un potentiel positif audit moyen (22) de protection afin d'établir un potentiel à travers la jonction p/n formée par le silicium (10) et la couche n (17).

9. Procédé selon la revendication 8, dans lequel ladite étape de revêtement desdites première et seconde surfaces (14, 15) consiste:

à faire croître une couche d'oxyde sur lesdites première et seconde surfaces (14, 15);

à déposer un nitrure de silicium (18, 19) sur ladite surface oxydée; et

à faire croître une couche d'oxyde sur lesdites couches de nitrure (18, 19).

10. Procédé selon la revendication 9, dans lequel le processus d'enlèvement dudit revêtement consiste:

à revêtir lesdites première et seconde surfaces de silicium d'une réserve photosensible;

à exposer la réserve photosensible sur les deux côtés du silicium pour définir les sections d'entrée (20) et de sortie (21) de la buse;

à éliminer par gravure les couches d'oxyde-nitrure-oxyde (18, 19) des sections (20, 21) définissant l'entrée (11) et la sortie (13) de la buse.

11. Procédé selon la revendication 10, dans lequel lesdites sections exposées de sortie (13) et d'entrée (11) ont des formes circulaires.

12. Procédé selon la revendication 8, dans lequel ladite solution de gravure est une solution de KOH.

13. Procédé selon la revendication 6, dans lequel ladite cavité et ledit passage sont concentriques.

14. Procédé pour former une structure de buse comprenant une ouverture dans une mince section de silicium monocristallin de type p, à orientation cristallographique, ayant des surfaces planes avant et arrière (14, 15), le procédé consistant:

à former une couche superficielle n (17) sur la surface arrière (15) de la section (10) de silicium;

à revêtir les deux surfaces avant et arrière (14, 15) de la section (10) de silicium, sauf les sections (20, 21) qui définissent l'entrée (11) et la sortie (13) de la buse, avec une matière (18, 19) qui résiste à la gravure;

à appliquer un revêtement protecteur (22) sur le côté de sortie de l'ouverture;  
à immerger la section (10) de silicium dans une solution de gravure;  
à immerger une cathode dans ladite solution;  
à appliquer un potentiel positif de commande à la couche n (17);  
à graver de façon anisotrope une cavité depuis le côté d'entrée de la buse jusqu'à la couche n (17) sur le côté de sortie de la buse;  
à enlever la section de silicium (10) de la solution de gravure;  
à enlever la couche protectrice (22) recouvrant

la zone de sortie de la buse;  
à revêtir le côté d'entrée d'une couche protectrice (24);  
à immerger de nouveau le corps (10) de silicium dans la solution de gravure; et  
à gravure de façon anisotrope un passage (13) depuis la surface arrière de la section (10) de silicium jusqu'à la cavité, à travers la couche n (17).  
15. Procédé selon la revendication 14, dans lequel ledit silicium monocristallin est orienté suivant les plans (100) ou (110).

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⑫ **NEW EUROPEAN PATENT SPECIFICATION**

④⑤ Date of publication of the new patent specification: **01.06.94 Bulletin 94/22**

⑤① Int. Cl.<sup>5</sup>: **B41J 2/135, B41J 2/16, B05B 1/00**

②① Application number: **85112882.7**

②② Date of filing: **11.10.85**

⑤④ **Silicon nozzle structures and method of manufacture.**

③⑩ Priority: **15.10.84 US 661005**

④③ Date of publication of application: **23.04.86 Bulletin 86/17**

④⑤ Publication of the grant of the patent: **16.01.91 Bulletin 91/03**

④⑤ Mention of the opposition decision: **01.06.94 Bulletin 94/22**

⑧④ Designated Contracting States: **BE DE FR GB IT NL**

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**EP 0 178 596 B2**

**Description****Technical Field**

5 Monocrystalline silicon bodies with passages.

**Background of the Invention**

10 In the prior art and specifically in US-A-3,921,916 it is suggested that a monocrystalline, crystallographically oriented silicon wafer may be selectively etched to form one or more reproducible channels of a specific form in the wafer body. The specific type of the channel described in that patent has a rectangular entrance cross-section which continues to an intermediate rectangular cross-section, smaller than the entrance cross-section, and then to an exit cross-section which has a shape other than rectangular. A channel of this specific type is established by either of two disclosed processes, both of which utilize a heavily doped p+ layer (patterned in the one process and unpatterned in the other) as an etchant barrier. In the two processes, a silicon wafer is heavily doped to place it near or at saturation from one major face to form the p+ etchant barrier. Thereafter, patterned anisotropic etching from the opposite major face proceeds until the p+ barrier is reached. The anisotropic etching results in a rectangular entrance cross-section and a rectangular intermediate cross-section defining a membrane smaller in size than the entrance cross-section.

20 In the application of one process, the etching process is continued from the entrance side until an opening is made through the membrane. The other process utilizes patterned isotropic etching from the opposite side (exit side) of the nozzle to complete a passage through the membrane to the intermediate cross-section.

Although these prior art processes may provide satisfactory ink jet nozzle structures, both of the described processes and the resulting structures have inherent problems. For example, due to inherent wafer thickness variations and isotropic etch nonuniformities, these processes require extensive mechanical and/or chemical polishing of both major surfaces of the wafer to improve dimensional control of the resulting nozzle structures. This is a costly processing step. Additionally, the nozzle structures produced by these processes have heavily saturated p+ regions surrounding the exit openings, and these regions tend to be brittle and thus subject to failure when exposed to high fluid pressures or pressure transients typically present in ink jet printing systems.

**Disclosure of Invention**

35 In accordance with the present invention, a standard commercially available semi-conductor wafer of crystallographically oriented, monocrystalline p-type silicon is used to produce a single fluid nozzle or an array of nozzles directly and without the need for mechanical or chemical polishing of the two major surfaces of the wafer by a process wherein a low saturation n surface layer is formed on at least one major surface of the wafer. Materials resistant to an anisotropic etchant, later employed, are then deposited on both surfaces of the wafer. Thereafter, aperture masks defining the entrance and exit areas of a nozzle are formed on these major surfaces and the exit area is coated with a material which is both resistant to an etching solution and which provides an electrical connection to the n layer. A cavity is anisotropically etched from the entrance area of the wafer through to the n layer at the exit side by immersing the wafer in a caustic etching solution. A potential applied across the p/n junction at the exit side of the wafer electro-chemically stops the etching action leaving a membrane having a thickness substantially equal to the n-layer.

45 A passage is then anisotropically etched through the membrane from the exit side to complete the nozzle structure.

**The Drawings**

- 50 Fig. 1 shows a perspective view of a portion of the nozzle structure in accordance with the present invention;  
 Fig. 2 is a cross-sectional view of the nozzle structure taken along line 2-2 of Fig. 1;  
 Fig. 3 through 8 illustrate sequential cross-sectional views of a silicon wafer processed in accordance with the present invention.

**Detailed Description**

65 In multi-nozzle ink jet printing systems utilizing nozzles made of semi-conductor material, some of the more important characteristics required of the nozzle are the uniformity in the size of each respective nozzle, spatial



distribution of the nozzles in an array, their resistance to cracking under the fluidic pressures encountered in the system, provision of an efficient mechanical impedance match between the fluid supply and the exit opening, as well as, their resistance to wear caused by the high velocity fluid flow through the nozzle structure.

Referring now to Fig. 1, there is shown a portion of the nozzle structure made in accordance with the present invention. Specifically a substrate 10 is shown having an array of uniform openings 11 therein. Each opening 11 starts with an initial, substantially square area and tapers to and terminates in a substantially square area smaller than the initial square area defining a membrane 12. As shown in Fig. 2, each membrane 12 in turn has an opening 13 extending therethrough which starts in a substantially square area smaller than the square area of each respective membrane 12 and terminates in a substantially square area larger than the starting square area of said opening. Both horizontal axes of the openings 13 in the membrane 12 are substantially aligned with the horizontal axes of each corresponding opening 11 in the main body of the wafer 10 by virtue of the wafer 10 crystallography.

Figs. 3 through 8 illustrate a sequence of process steps for production of an aperture in a single crystal silicon wafer 10 for forming one fluid nozzle or an array of nozzles. It is to be understood that the following process steps may be used in a different sequence and that other film materials for performing the same functions described below may be used. Furthermore, film formation, size, thickness and the like, may also be varied. The wafer 10 is of single crystal (100) oriented p type silicon with electrical resistivity of 0.5 to 100 ohm-cm, approximately 495  $\mu\text{m}$  to 515  $\mu\text{m}$  (19.5 to 20.5 mils) thick having front 14 and back 15 surfaces. The (100) planes are parallel to surfaces 14 and 15. As shown in Fig. 3, phosphorous is diffused into the front 14 and back 15 surfaces of the silicon wafer 10 to a depth of about 5  $\mu\text{m}$  forming n type layers 16 and 17. As will become obvious later only one diffused layer is required to form a nozzle structure by the process (exit side). The diffusion is accomplished in a well-known manner by having a gas mixture containing 0.75 %  $\text{PH}_3$ , 1 %  $\text{O}_2$ , and the make-up of Ar and  $\text{N}_2$  flow for 30 minutes past the silicon wafer 10 which is maintained at 950°C. This is followed by a long drive-in period (1050°C for 22 hours) to achieve a thick layer (about 5 microns). Since the final concentration of phosphorous in the n layers 16 and 17 is very low, this diffusion step introduces very little stress into the silicon wafer 10, and consequently the silicon structure retains its strength.

Next as shown in Fig. 4, both front 14 and back 15 surfaces of the wafer 10 are coated with a protective material such as LPCVD silicon nitride forming layers 18 and 19 which can resist a long etching period in a caustic (KOH) solution. One of the ways to accomplish this is to utilize a low pressure chemical vapor deposition of silicon nitride deposited at about 800°C. Oxide layers (not shown) less than 0.5  $\mu\text{m}$  thick may be grown on both sides of layers 18 and 19 to reduce the effect of stress between nitride and silicon and to improve adhesion of photoresist to nitride. To promote ease of photoshaping it is recommended that the wafer 10 when procured have its back surface 15 etched in an acidic rather than caustic solution.

Thereafter, masks are prepared corresponding to the desired entrance 20 and exit 21 areas of the nozzle. The masks for both entrance 20 and exit 21 areas are made circular in shape since the openings in the silicon wafer 10 defined by circular masks will etch out to squares parallel to the 100 planes, each square circumscribing its respective circle. Use of circular masks eliminates possible error due to the theta misalignment which may occur when a square shaped mask is used. The silicon nitride layers 18 and 19 are photoshaped simultaneously on both sides using a two-sided photospinner (not shown) and a two-sided aligner (not shown). The resulting structure after etching away of portions of layers 18 and 19 defining the entrance 20 and exit 21 areas, is shown in Fig. 5.

The exit area 21 is then protected from the etching solution by covering it with a metallic layer 22, as shown in Fig. 6, or by use of a hermetic mechanical fixture (not shown). Thereafter the wafer is submerged in a hot (80-85°C) KOH solution (not shown) and a potential is placed across the p/n junction at the back side 15 by connecting the positive side of an electrical power source (not shown) with the metallic layer 22 protecting the exit area 21. Other alkaline etch solutions such as metal hydroxides of the Group I-A elements of the Periodic Table, for example, NaOH,  $\text{NH}_4\text{OH}$ , or others, may be used. The use of electrochemically controlled thinning process for semi-conductors is well-known in the art and is described in detail in US-A-3,689,389.

The opening 11 in the monocrystalline silicon wafer 10 is etched anisotropically until the diffused layer 17 at the back side 25 is reached, at which time the etching action stops due to an oxide layer (not shown) which is caused to grow at the p/n junction due to the applied potential across the junction. It is well known in the art that the (111) plane is a slow etch plane in monocrystalline silicon material when a KOH etching solution is used. Thus, the etching step produces a pyramidal opening in the wafer 10 which opening truncates in a membrane 12 when it encounters the electrochemical etch barrier set up at the silicon and diffused layer 17 interface (p/n junction).

Thereafter, the wafer 10 is removed from the etching solution, the protective metallic layer 22 and associated electrical connection on the exit side are removed, and the entrance side 20 is protected from the etching solution usually by a layer 24 formed by air oxidation. The wafer 10 is then re-submersed into the etching

solution and a pyramidal passage is etched anisotropically from the back surface 15 to form the exit opening 13. The resulting structure is shown in Fig. 7.

If desired, the protective coatings 18, 19 and 24 are then removed leaving a completed pure silicon nozzle structure as shown in Fig. 8. Typically the initial opening of the entrance 20 is about 890  $\mu\text{m}$  (35 mils) wide and the smallest portion of the exit opening 13 is about 38  $\mu\text{m}$  to 102  $\mu\text{m}$  (1.5 to 4 mils) wide.

Since the etch rate perpendicular to the (111) planes is very low compared to the vertical etch rate (100), overetch does not mitigate against the high accuracy defined by the exit mask. To prevent ink from wetting the surface of the wafer on the exit side, the back surface 15 of the wafer 10 may be coated with a material of low surface energy such as Teflon.

#### Claims

1. A nozzle comprising a nozzle body (10) formed of a monocrystalline p-type silicon material having a rectangular entrance aperture (11) of a first cross-sectional area which tapers to a second rectangular cross-sectional area which is smaller than the first cross-sectional area of said entrance aperture; and a membrane (12) of semi-conductor material formed within said second cross-sectional area and having an exit aperture;
  - said membrane (12) being of
    - n-type monocrystalline silicon having a thickness of 10 micrometers or less and has a rectangular exit aperture (13) therein, said exit aperture (13) having a first cross-sectional area which is smaller than the second cross-sectional area of the entrance aperture (11) and said exit aperture tapering from said second cross-sectional area of said entrance aperture to a second cross-sectional area of said exit aperture, said exit aperture first cross-sectional area being smaller than said second cross-sectional area thereof, said cross-sections being substantially parallel to the (100) planes of the monocrystalline silicon and said entrance and exit apertures being substantially concentric.
2. A process for forming a nozzle structure comprising an aperture in a section (10) of crystallographically oriented, p-type, monocrystalline silicon having first (15) and second (14) major surfaces;
  - said process comprising the steps of
    - forming a n surface layer (17) on the first major surface (15) of the silicon section (10);
    - coating said first and second surfaces (14, 15) of the silicon section (10) with a material (18, 19) which resists etching;
    - removing said coating from said first and second surfaces in the area (20, 21) defining the entrance (11) and exit (13) of the nozzle respectively;
    - protecting the exit area (21) from the etching solution;
    - establishing an electrochemical barrier at the silicon n layer interface;
    - anisotropically etching a cavity from the second surface (14) of the silicon section to said n layer (17); and
    - anisotropically etching a passage (13) through said n layer (17) from the first surface (15) of the silicon section (10).

#### Patentansprüche

1. Düse mit einem Düsenkörper (10), der aus einem monokristallinen p-Siliziummaterial geformt ist und der eine Eingangsöffnung (11) aufweist mit einer ersten rechteckigen Querschnittsfläche, die konisch zuläuft zu einer zweiten rechteckigen Querschnittsfläche, die kleiner ist als die erste Querschnittsfläche, und einer Membran (12) aus Halbleitermaterial, die in der zweiten Querschnittsfläche erzeugt ist und eine Ausgangsöffnung (13) aufweist, wobei die Membran (12) aus n-Silizium mit einer Dicke von 10 Mikrometer oder weniger besteht und eine rechteckige Ausgangsöffnung (13) besitzt, die Ausgangsöffnung (13) eine erste Querschnittsfläche aufweist, die kleiner ist als die zweite Querschnittsfläche der Eingangsöffnung (11), die Ausgangsöffnung (13) von der zweiten Querschnittsfläche der Eingangsöffnung zu einer zweiten Querschnittsfläche der Ausgangsöffnung konisch zuläuft, die erste Querschnittsfläche der Ausgangsöffnung kleiner als die zweite Querschnittsfläche der Ausgangsöffnung ist, die Querschnittsflächen im wesentlichen parallel zu den (100)-Ebenen des monokristallinen

stallin n-Siliciums verlaufen, und die Eingangs- und Ausgangsöffnung im wesentlichen konzentrisch sind.

- 5 2. Verfahren zur Herstellung einer Düsenstruktur, die eine Öffnung in einem Abschnitt (10) aus kristallographisch orientiertem, monokristallinem p-Silicium mit einer ersten (15) und einer zweiten (14) Hauptfläche enthält,  
mit den Verfahrensschritten:  
Herstellen einer n-Oberflächenschicht (17) auf der ersten Hauptfläche (15) des Siliciumabschnitts (10);  
10 Beschichten der ersten und zweiten Hauptfläche (14, 15) des Siliciumabschnitts (10) mit einem gegen Ätzen beständigen Material (18, 19);  
Entfernen der Beschichtung von der ersten und zweiten Hauptfläche in den Bereichen (20, 21), die den Eingang (11) bzw. Ausgang (13) der Düse definieren;  
Schützen des Ausgangsbereiches (21) vor der Ätzlösung; Herstellen einer elektrochemischen Barriere  
15 an der n-Schicht-Grenzfläche,  
anisotropes Ätzen eines Hohlraumes (11), ausgehend von der zweiten Hauptfläche (14) des Siliciumabschnitts bis zu der n-Schicht (17); und  
anisotropes Ätzen einer Öffnung (13) durch die n-Schicht (17) von der ersten Hauptfläche (15) des Siliciumabschnitts (10) aus.

## Revendications

- 25 1. Buse comprenant un corps (10) de buse formé d'une matière du type silicium monocristallin de type p présentant une ouverture d'entrée rectangulaire (11) d'une première coupe transversale qui s'effile jusqu'à une seconde coupe transversale rectangulaire qui est plus petite que la première coupe transversale de ladite ouverture d'entrée ; et  
une membrane (12) en matière semi-conductrice formée à l'intérieur de ladite seconde coupe transversale et ayant une ouverture de sortie ;  
30 ladite membrane (12) étant en silicium mono-cristallin de type n ayant une épaisseur de 10 micromètres ou moins et présentant une ouverture rectangulaire (13) de sortie, ladite ouverture (13) de sortie ayant une première coupe transversale qui est plus petite que la seconde coupe transversale de l'ouverture d'entrée (11) et ladite ouverture de sortie s'amincissant de ladite seconde coupe transversale de ladite ouverture d'entrée jusqu'à une seconde coupe transversale de ladite ouverture de sortie, ladite  
35 première coupe transversale de l'ouverture de sortie étant plus petite que sa seconde coupe transversale, lesdites coupes transversales étant sensiblement parallèles aux plans (100) du silicium monocristallin et lesdites ouvertures d'entrée et de sortie étant sensiblement concentriques.
- 40 2. Procédé pour former une structure de buse comprenant une ouverture dans une partie (10) de silicium monocristallin de type p, à orientation cristallographique, présentant des première (15) et seconde (14) surfaces principales ;  
ledit procédé comprenant les étapes qui consistent  
à former une couche superficielle n (17) sur la première surface principale (15) de la partie (10) en silicium ;  
45 à revêtir lesdites première et seconde surfaces (14, 15) de la partie (10) en silicium d'une matière (18, 19) qui résiste à la gravure ;  
à enlever ledit revêtement desdites première et seconde surfaces dans la section (20, 21) définissant l'entrée (11) et la sortie (13) de la buse, respectivement ;  
à protéger la section de sortie (21) de la solution de gravure ;  
50 à établir une couche d'arrêt électrochimique à l'interface du silicium et de la couche n ;  
à graver de façon anisotrope une cavité depuis la seconde surface (14) de la partie en silicium jusqu'à ladite couche n (17) ; et  
à graver de façon anisotrope un passage (13) à travers ladite couche n (17) depuis la première surface (15) de la partie (10) en silicium.

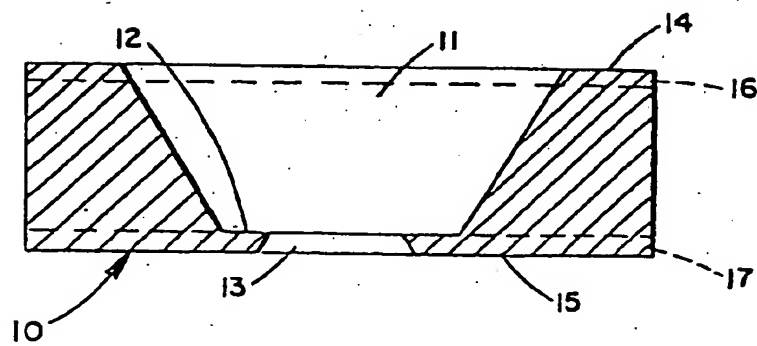
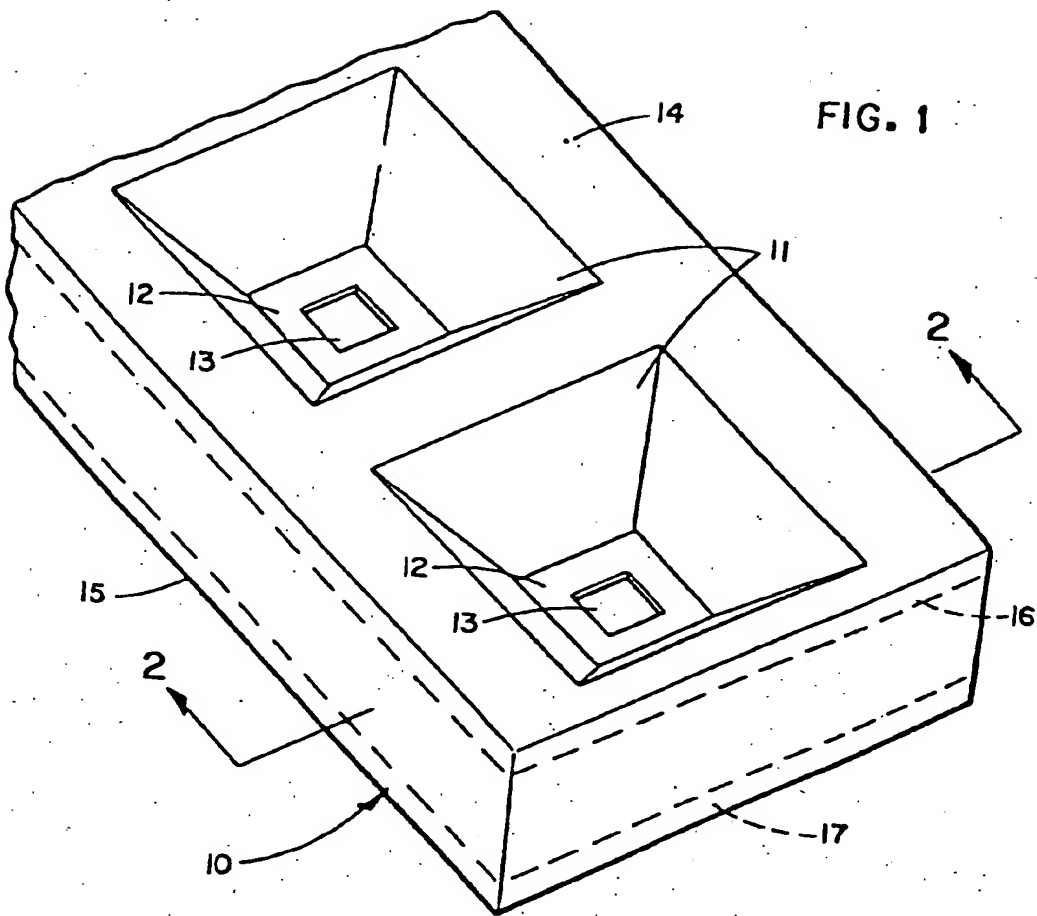


FIG. 3

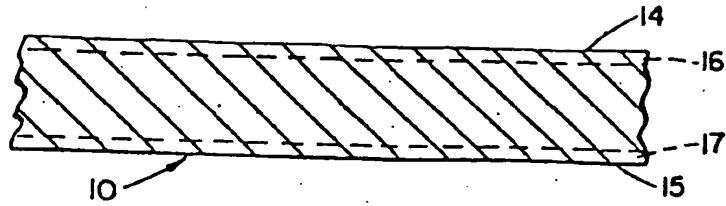


FIG. 4

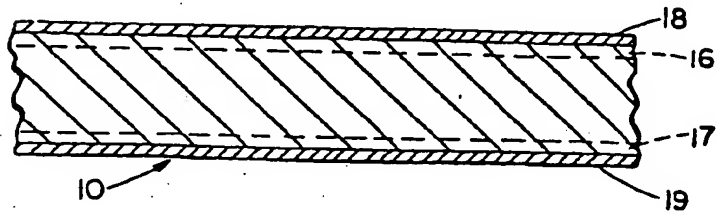


FIG. 5

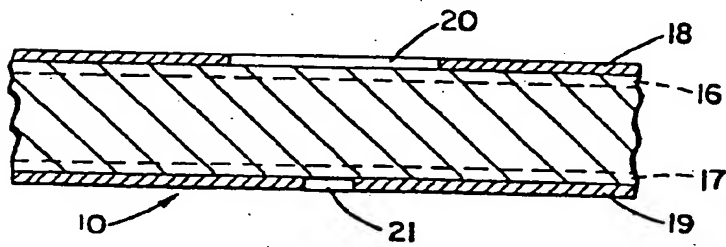


FIG. 6

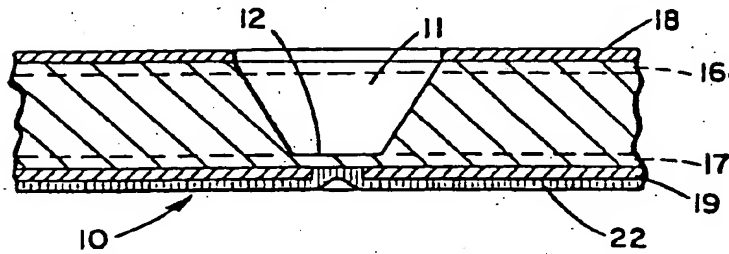


FIG. 7

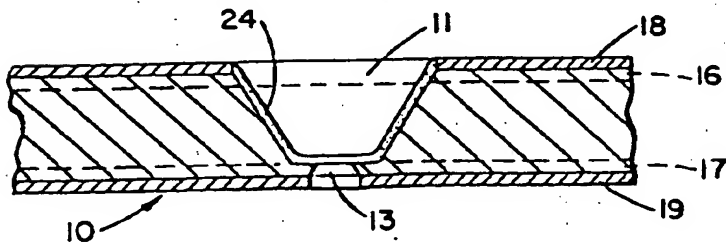
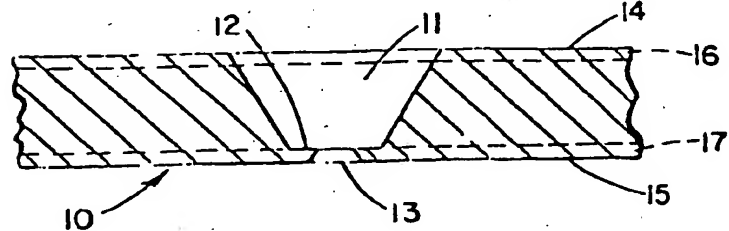


FIG. 8



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